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TITLE: Study of benzofuran. V. Structure of the diketones obtained from the acylation of 2-ethyl-3-acylbenzofurans
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AB cf. CA 54, 24632a; 55, 505b. 2-Ethyl-3-benzoyl- and 2-ethyl-3-anisoylbenzofurans have been acetylated under Friedel-Crafts conditions with excess AlCl3. Substitution occurred first at the 6-position, next at the 5-position. 2-Acylbenzofurans were not acetylated under the same conditions. To a solution of 1 mole 2-ethyl-3-benzoylbenzofuran (I) and 2 moles AcCl in 700 cc. CS2 was gradually added 2.5 moles AlCl3. The mixture was kept 24 hrs., then decomposed and purified to yield 4.5% I and a mixture, b. 240-60°, which on fractional crystallization from EtOH gave 40% 2-ethyl-3-benzoyl-6-acetylbenzofuran (II), m. 118.5° and 28% 2-ethyl-3-benzoyl-5-acetylbenzofuran (III), m. 68° (ligroine). Similarly, 2-ethyl-3-(4-methoxybenzoyl)benzofuran (IV) with AcCl gave 45% 2-ethyl-3-(4-methoxybenzoyl)-6-acetylbenzofuran (V), m. 118.5-19°, and 7% 2-ethyl-3-(4-methoxybenzoyl)-5-acetylbenzofuran (VI), m. 100-1° (EtOH, then ligroine-C6H6). V was demethylated by refluxing 20 min. with pyridine-HCl to 2-ethyl-3-(4-hydroxybenzoyl)-6- acetylbenzofuran, m. 214-15° (EtOH, or C6H6-ligroine). VI did not respond to similar treatment. NaOH degradation of II gave 4,2-Ac(HO)C6H3CH2COPh (VII), m. 212-13°, BzOH, and 4,2-Ac(HO)C6H3CH2COEt (VIII). Treatment of III with NaOH gave BzOH, 5,2-Ac(HO)C6H3CH2COPh (IX), m. 179°, and 5,2-Ac(HO)C6H3CH2COEt (X). Similarly, V with NaOH gave 4,2-Ac(HO)C6H3CH2COC6H4OMe-p (XI), m. 211°, anisic acid, and VIII. NaOH degradation of VI yielded 5,2-Ac(HO)C6H3CH2COC6H4OMe-p (XII), m. 165-7°, anisic acid, and X. The mixture of VII and VIII obtained from the NaOH degradation of II was methylated with MeI to yield 31% 4,2-Ac(MeO)C6H3CH2COEt (XIII), b17 201-4°, n22 1.5390, and 25.5% 4,2-Ac(MeO)C6H3CH2COPh (XIV), b17 247-8°, m. 66° (ligroine). Methylation of the mixture of VIII and XI gave 11% XIII and 51.1% 4,2-Ac(MeO)C6H3CH2COC6H4OMe-p (XV), b16 275-8°, m. 69-70° (ligroine-20% cyclohexane). Heating VIII in EtOH saturated with HCl gave 90% 2-ethyl-6-acetylbenzofuran (XVI), b12 163-5°, n20.5 1.5845, m. 20-2°; oxime m. 93.5° (dilute Et2O or ligroine). NaOBr treatment of XVI gave 33% 2-ethyl-6- benzofurancarboxylic acid, m. 171-2°. XVI was reduced by N2H4 in (CH2OH)2 to 2,6-diethylbenzofuran, b15 126.5°, n22.5 1.5415. In the same way, VII, heated in EtOH saturated with HCl gave 2-phenyl-6-acetylbenzofuran, m. 103-4° which was reduced by N2H4 to 2-phenyl-6-ethylbenzofuran, m. 52-3° (EtOH). XI heated in EtOH saturated with HCl gave 80% 2-(4-methoxyphenyl)-6-acetylbenzofuran, m. 147° (EtOH-C6H6), which was demethylated to 2-(4-hydroxyphenyl)-6-

acetylbenzofuran, m. 228° (EtOH or C6H6) and reduced by N2H4 to 2-(4-hydroxyphenyl)-6-ethylbenzofuran, m. 170-1° (dilute EtOH), b14 244-7°. X heated in EtOH saturated with HCl gave 2-ethyl-5-acetylbenzofuran, b23 179-80°, m. 44-5°; oxime m. 83°. The latter, treated with NaOBr gave 2-ethyl-5- benzofurancarboxylic acid, m. 165° (dilute EtOH). Cyclization of IX yielded 2-phenyl-5-acetylbenzofuran, m. 160° (EtOH), which was reduced by N2H4 in (CH2OH)2 to 2-phenyl-5-ethylbenzofuran, m. 76° (EtOH), b22 212-14°. XII was also cyclized (EtOH-HCl) to give 68% 2-(4-methoxyphenyl)-5-acetylbenzofuran, m. 170° (EtOH-C6H6), which was simultaneously reduced and demethylated by N2H4 to 2-(4-hydroxyphenyl)-5-ethylbenzofuran, m. 186° (dilute EtOH or C6H6). 5-Ethylsalicylaldehyde, b16 115-16°, n21.5 1.5545, was treated with ClCH2-COMe and KOH in EtOH to yield 57% 2-acetyl-5-ethylbenzofuran, b15 163-4°, m. 33-4° (EtOH), which was reduced to 2,5-diethylbenzofuran (XVII), b14 122-4°, n18 1.5430. XVII was benzoylated (ClCOPh, SnCl4, C6H6) in 67% yield to give 2,5-diethyl-3-benzoylbenzofuran (XVIII), b13 220-2°, n20 1.5995. Anisoylation of XVII gave 2,5-diethyl-3-(4-methoxybenzoyl)benzofuran (XIX), b12 247°, m. 32-3° (EtOH). The latter was demethylated to 2,5-diethyl-3-(4-hydroxybenzoyl)benzofuran, b4 272-3°, m. 135-6° (C6H6). NaOH degradation of XVIII followed by HCl-EtOH recyclization gave 43% PhCO2H, 22.5% XVII, and 40% 2-phenyl-5-ethylbenzofuran. Similar treatment of XIX gave 29.5% anisic acid, 2-(4-methoxyphenyl)-5-ethylbenzofuran, m. 135°, 5,2-Et(HO)C6H3CH2COC6H4OMe-p, m. 118°. Starting with 3-ethylsalicylaldehyde, b28 117-18°, a similar series of reactions was carried out giving 2-acetyl-7-ethylbenzofuran, b19 159-61°, m. 54.5° (EtOH), 2,7-diethylbenzofuran (XX), b20 124-5°, n22 1.5410, and 2,7-diethyl-3-benzoylbenzofuran, b15 228-31°, n21.5 1.6038. NaOH degradation of the latter gave BzOH, XX, and 2-phenyl-7-ethylbenzofuran, b20 220-3°, n24 1.6210.

CC 10G (Organic Chemistry: Heterocyclic Compounds)

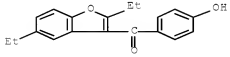
IT Acylation
(of 3-acyl-2-ethylbenzofurans, structure of diketones from)

IT Ketones
(structure of di-, from acylation of 3-acyl-2-ethylbenzofurans)

IT 3131-63-3, Benzofuran, 2-ethyl-
(3-acyl derivs., diketones from acylation of)

IT 5896-26-4P, Ketone, 2-ethyl-6-benzofuranyl methyl 5896-49-1P, Benzofuran, 2,6-diethyl- 27408-42-0P, Ketone, 2-ethyl-6-benzofuranyl methyl, oxime 28089-83-0P, Ketone, methyl 2-phenyl-6-benzofuranyl 59664-03-8P, Ketone, 7-ethyl-2-benzofuranyl methyl 91495-47-5P, Benzofuran, 2,5-diethyl- 93021-68-2P, Benzofuran, 5-ethyl-2-phenyl- 94066-54-3P, 2,4'''-Biacetophenone, 3'''-hydroxy- 94302-86-0P, Ketone, 2,5-diethyl-3-benzofuranyl p-hydroxyphenyl 95485-40-8P, Ketone, 2-ethyl-5-benzofuranyl methyl 100612-37-1P, Acetophenone, 3'-methoxy-4'-(2-oxobutyl)- 101278-17-5P, Ketone, 2-(p-hydroxyphenyl)-6-benzofuranyl methyl 101594-95-0P, Acetophenone, 2-(5-ethyl-2-hydroxyphenyl)-4'-methoxy- 101596-59-2P, Benzofuran,

5-ethyl-2-(p-methoxyphenyl)- 101894-27-3P, Benzofuran,
6-acetyl-2-ethyl-3-p-hydroxybenzoyl- 101894-27-3P, Phenol,
p-(6-acetyl-2-ethyl-3-benzofuranylcarbonyl)- 102158-98-5P,
Ketone,
2,5-diethyl-3-benzofuranyl p-methoxyphenyl 103152-25-6P,
2,4'''-Biacetophenone, 3'''-methoxy- 103988-06-3P, 5-
Benzofurancarboxylic acid, 2-ethyl- 105207-89-4P, Acetophenone,
4'-hydroxy-3'-(2-oxobutyl)- 105208-20-6P, Acetophenone,
3'-hydroxy-4'-(2-oxobutyl)- 105909-84-0P, Ketone, 2-ethyl-5-
benzofuranyl
methyl, oxime 106989-39-3P, Ketone, 5-ethyl-2-benzofuranyl
methyl
108838-38-6P, 2,3'''-Biacetophenone, 4'''-hydroxy- 108840-63-7P,
Benzofuran, 6-ethyl-2-phenyl- 108840-64-8P, Benzofuran,
7-ethyl-2-phenyl- 108840-82-0P, Phenol, p-6-ethyl-2-
benzofuranyl-
108842-68-8P, Phenol, p-5-ethyl-2-benzofuranyl- 108980-53-6P,
Ketone,
2-(p-methoxyphenyl)-6-benzofuranyl methyl 108983-46-6P, Ketone,
2-(p-methoxyphenyl)-5-benzofuranyl methyl 109155-31-9P,
2,4'''-Biacetophenone, 3'''-hydroxy-4'-methoxy- 109156-66-3P,
2,3'''-Biacetophenone, 4'''-hydroxy-3'-methoxy- 109395-02-0P,
2,4'''-Biacetophenone, 3''',4'-dimethoxy- 109614-26-8P,
Benzofuran,
6-acetyl-3-benzoyl-2-ethyl- 109614-90-6P, Benzofuran,
5-acetyl-3-benzoyl-2-ethyl- 109688-24-6P, Ketone, 2,7-diethyl-3-
benzofuranyl phenyl 109690-79-1P, Ketone, 2,5-diethyl-3-
benzofuranyl
phenyl 109893-46-1P, 1-Propanone, 1-[5(or 6)-acetyl-2-ethyl-3-
benzofuranyl]- 109936-61-0P, Benzofuran, 6-acetyl-3-p-anisoyl-2-
ethyl-
109938-44-5P, Benzofuran, 5-acetyl-3-p-anisoyl-2-ethyl- 121045-
41-8P,
Ketone, methyl 2-phenyl-5-benzofuranyl 857020-75-8P,
6-Benzofurancarboxylic acid, 2-ethyl- 857021-42-2P, Benzofuran,
2,7-diethyl-
RL: PREP (Preparation)
(preparation of)
IT 94302-86-0P, Ketone, 2,5-diethyl-3-benzofuranyl p-hydroxyphenyl
101894-27-3P, Benzofuran, 6-acetyl-2-ethyl-3-p-hydroxybenzoyl-
RL: PREP (Preparation)
(preparation of)
RN 94302-86-0 CAPLUS
CN Ketone, 2,5-diethyl-3-benzofuranyl p-hydroxyphenyl (6CI, 7CI) (CA
INDEX
NAME)



RN 101894-27-3 CAPLUS

CN Phenol, p-(6-acetyl-2-ethyl-3-benzofuranylcarbonyl)- (6CI) (CA INDEX NAME)